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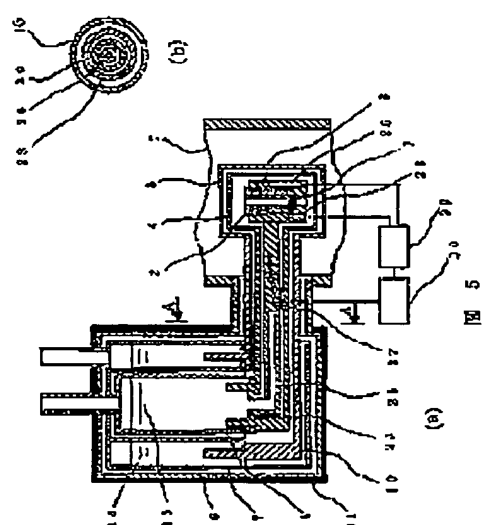
(54)【発明の名称】 試料冷却装置

(57)【要約】
【課題】 電子顕微鏡等の試料冷却装置の極低温化と温度を補償すること。
【解決手段】 液体窒素貯留容器17の内側に液体ヘリウム貯留容器18を設けた二槽構造の冷媒貯留容器16とし、液体ヘリウム貯留容器18に結合され独立した二つの第一、第二熱伝導部材23、26と液体窒素貯留容器17に結合された第三の熱伝導部材20を第一熱伝導部材23の外側に各々略筒状に包含し熱伝導と熱シールドを兼ね備える。第一熱伝導部材23は中空とし冷媒が流入するように形成するとともに熱伝導面積をピエゾ素子27で可変可能とする。試料1もしくは試料ホルダー2あるいは試料1もしくは試料ホルダー2を積載する冷却部材3にヒータ25および温度センサー28を設け、温度制御装置29とピエゾ素子駆動装置30を閉ループで制御することで任意の温度に設定制御し温度を補償する。



【特許請求の範囲】

【請求項１】電子顕微鏡等の鏡体内に設置される試料もしくは試料ホルダーを搭載する試料ステージを複数の熱シールド層で包含し、その試料ステージと各々の熱シールド層を冷却するための冷媒貯留容器と、両者をつなぐ熱伝導部材からなる試料冷却装置において、冷媒貯留容器に熱伝導部材の一端が結合され、当該熱伝導部材の熱伝導部を複数の略筒状に分割し、複数の熱シールド層を兼ね、各々独立した熱伝導部材とすることを特徴とする試料冷却装置。

【請求項２】前記冷媒貯留容器と熱伝導部材の結合おいて、冷媒貯留容器に熱伝導部材の一端が結合され、かつ、当該熱伝導部材を略筒状の複数の熱伝導部で構成するように配置し、複数の熱シールド層を兼ね、各々独立した熱伝導部材とすることを特徴とする請求項１記載の試料冷却装置。

【請求項３】前記冷媒貯留容器と熱伝導部材の結合おいて、冷媒貯留容器に熱伝導部材の一端が冷媒貯留容器の中に突出し、冷媒と熱伝導部材とが直接接触ように構成したことを特徴とする請求項１記載の試料冷却装置。

【請求項４】前記冷媒貯留容器と熱伝導部材の結合おいて、熱伝導部材を中空とし冷媒貯留容器に充填された冷媒が熱伝導部材の中空部に流入し、冷媒が充填されるように構成したことを特徴とする請求項１記載の試料冷却装置。

【請求項５】前記試料もしくは試料ホルダーを冷却する第一熱伝導部材と第一熱伝導部材の外側に略筒状に設けられた第二熱伝導部材を１つの液体ヘリウム冷媒貯留容器に結合し、第二熱伝導部材の外側に略筒状に設けられた第三熱伝導部材を液体窒素冷媒貯留容器に結合するように構成し、熱シールド層を設けたことを特徴とする請求項１記載の試料冷却装置。

【請求項６】前記熱伝導部材を任意の位置で熱伝導面積を可変にすることにより熱伝導熱量を制御し、かつ、試料および試料ホルダーまたは前記試料および試料ホルダー搭載冷却部材に入熱機能および温度検出機能を設け、試料もしくは試料ホルダーの温度を制御補償することを特徴とする請求項１又は２記載の試料冷却装置。

【請求項７】前記熱伝導面積の可変部に、ピエゾ素子を設け、ピエゾ素子駆動により前記熱伝導面積可変部の熱伝導面積を制御し、かつ、試料および試料ホルダーまたは前記試料および試料ホルダー搭載冷却部材に入熱機能および温度検出機能を設け、試料もしくは試料ホルダーの温度を補償することを特徴とする請求項６記載の試料冷却装置。

【請求項８】前記熱伝導部材を任意の位置で分割し、前記熱伝導部材の分割部熱伝導面間の熱接触時間を制御し、かつ、試料および試料ホルダーまたは前記試料および試料ホルダー搭載冷却部材に入熱機能および温度検出機能を設け、試料もしくは試料ホルダーの温度を制御補償することを特徴とする請求項１又は２記載の試料冷却装置。

【請求項９】前記熱伝導部材の分割部に、ピエゾ素子を設け、ピエゾ素子駆動により前記熱伝導部材の分割部熱伝導面間の熱接触時間を制御し、かつ、試料および試料ホルダーまたは前記試料および試料ホルダー搭載冷却部材に入熱機能および温度検出機能を設け、試料ホルダーの温度を補償することを特徴とする請求項７記載の試料冷却装置。

【請求項１０】前記液体ヘリウム冷媒貯留容器の外側に液体窒素冷媒貯留容器を設け、液体窒素冷媒貯留容器に熱接触した熱シールド部材で液体ヘリウム冷媒貯留容器包含し、液体ヘリウム冷媒貯留容器および液体窒素冷媒貯留容器を包含する冷媒容器に断熱支持固定し、冷媒容器外周に制振材を設け冷媒の蒸発による振動および冷媒容器外面より受ける音波等の空気振動を低減することを特徴とする請求項５記載の試料冷却装置

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は電子顕微鏡等の真空容器内に試料を設置する装置と組み合わせて使用する極低温試料冷却装置に関するものである。

【０００２】

【従来の技術】従来の電子顕微鏡等の極低温試料冷却装置として、例えば、特開６３－３２８４７号公報に記載のように、５Ｋ以下の極低温領域まで試料冷却可能な装置がある。

【０００３】図１は従来の電子顕微鏡等の極低温試料冷却装置の構成を示したものである。

【０００４】試料１もしくは試料ホルダ２と熱接触して極低温に冷却する第一冷却部材３と第一冷却部材３を略筒状に取り囲み熱シールドを行う第二および第三の冷却部材４、５で構成された試料ステージ６を電子顕微鏡鏡体７内に設置し、電子顕微鏡鏡体７外に互いに独立で、前記第一冷却部材３を冷却する第一の液体ヘリウム貯留容器８と、熱シールドを行う第二および第三の冷却部材４、５を冷却する第二の液体ヘリウム貯留容器９および液体窒素貯留容器１０が設置される。第一および第二の液体ヘリウム貯留容器８、９および液体窒素貯留容器１０が各々第一の冷却部３および第二、第三の冷却部材４、５とそれぞれ、第一熱伝導部材１１および第二、第三の熱伝導部材１２、１３で結合され極低温まで試料の冷却を可能にしていた。このように、従来の極低温試料冷却装置では熱伝導および熱輻射により低温部に流入する熱量は、それと接触または相対する高温部の温度が低い程少ないことから試料１および試料ホルダー２と室温部分との間に約１００Ｋに冷却された液体窒素冷却部材５を介在させ、さらに、１００Ｋ以上の液体窒素冷却部材５と数Ｋの液体ヘウム冷却部材３とが１００度程度の温度差を持って接触または相対しないようにするため、

試料１および試料ホルダー２を冷却する液体ヘウム冷却部材３と液体窒素冷却部材５との間に第二の液体ヘウム冷却部材４を介在させて，試料１および試料ホルダー２を囲う三重熱シールド構造とし、上記三つの冷却部材を三つの冷媒貯留容器からそれぞれ三つの熱伝導部材を介して冷却する構造となっていた。しかし、この方式では，液体窒素貯留容器が一槽と液体ヘリウム貯留容器が二槽必要となり，熱伝導および熱輻射により低温部に流入する熱量の変化や、試料温度をヒータ等の入熱により任意に設定する場合には，液体ヘリウムの蒸発潜熱は液体窒素に比べて１／１０と小さく，僅かな熱流入によって液体ヘリウムの消費量が大きく左右される。このことから，従来の液体ヘリウム貯留容器が二槽方式では各々の貯留容器の液体ヘリウム消費量が試料冷却の実施条件により異なることとなり，試料冷却可能（試料冷却実験）時間は二槽の液体ヘリウムの内の早く消費される側の液体ヘリウムによって決定されてしまう。このことは，高価な液体ヘリウムの有効活用ができず，液体ヘリウムを大量に消費することとなり経済性にも欠ける。また，液体ヘリウムを貯留容器入れる作業が二度となり作業性にも欠ける。当然貯留容器の製作費も三つの貯留容器となり、冷媒容器が大型化するためため製造価格が上がる等の問題点があった。さらに、冷媒の蒸発による振動および冷媒容器外面より受ける音波等の空気振動が熱伝導部材を介して試料もしくは試料ホルダに伝達され電子顕微鏡等の性能を低下させていた。

【０００５】

【発明が解決しようとする課題】上記従来技術の極低温試料冷却装置では、冷媒と極低温試料冷却装置の冷却特性により冷却到達温度が決定され、高温側へ任意の温度に設定制御する場合、ヒータ等により試料もしくは試料ホルダーまたは積載する熱伝導部材に入熱を行うと冷媒の蒸発を早めることとなり，必然的に試料冷却保持時間が短縮されるばかりか高価な冷媒を大量に消費することになる。このため冷媒の消費を低減し経済性の向上を図る必要がある。また、液体ヘリウムの蒸発潜熱は液体窒素に比べて１：１０と小さく僅かな熱流入によって二つの貯留容器の液体ヘリウムの消費量が異なり、二つの貯留容器の一方の液体ヘリウムの消費によって試料冷却保持時間が制限され二つの貯留容器に充填した液体ヘリウムが有効活用されない。

【０００６】さらに、液体窒素貯留容器が一槽と液体ヘリウム貯留容器が二槽の冷媒容器構成になっていることから貯留容器に冷媒を充填するのに多大の時間を要するために、冷媒を充填する作業時間を短縮する必要がある。

【０００７】

【課題を解決するための手段】本発明は、極低温試料冷却装置の冷媒貯留容器を液体窒素貯留容器とその内側に設けられた液体ヘリウム貯留容器の二槽構造とし，液体窒素貯留容器に結合された熱伝導部材の内側に液体ヘリウム貯留容器に結合された熱伝導部材を設け、この液体ヘリウム貯留容器に結合された熱伝導部の他端を二つに分割，または独立した二つの熱伝導部とし，熱伝導部を三層構造にする。さらに，試料もしくは試料ホルダを冷却する熱伝導部材の熱伝導面積を可変に、あるいは，熱伝導部材を熱伝導面で分割し、分割部熱伝導面間の熱接触時間を制御することにより液体ヘリウムの消費量を制限し，試料および試料ホルダーまたは前記試料および試料ホルダー搭載冷却部材に入熱機能および温度検出機能を設け、試料もしくは試料ホルダーの温度を制御補償することが実現される。

【０００８】液体窒素で冷却された熱伝導部材の内側に液体ヘリウムで冷却された二つの熱伝導部材を三層構造に包含構成し熱シールド層を設け、中間層に流入する熱を液体ヘリウムで吸熱する。さらに，試料もしくは試料ホルダを冷却する冷却部材にヒータおよび温度センサーを設置し、熱伝導部材の熱伝導面積を可変に、あるいは，熱伝導部材を熱伝導面で分割し，分割部熱伝導面間の熱接触時間を制御することで高温側へ任意の温度に設定制御し温度を補償する。これにより，液体ヘリウムの消費量を低減する。

【０００９】

【発明の実施の形態】以下，本発明の一実施例を図を用いて説明する。

【００１０】図２（ａ）は本発明の実施例で二槽構造の冷媒貯留容器に熱伝導部材が突出し結合され、低温側の冷媒貯留容器に結合された熱伝導部材の他端を二つの略筒状に分割，三層の熱伝導と熱シールドを兼ねた極低温試料冷却装置の構成図を，（ｂ）は（ａ）の熱伝導部材のＡ－Ａ断面図を示す。試料１もしくは試料ホルダ２は本冷却装置と熱接触して極低温に液体窒素１４および液体ヘリウム１５を用いて熱伝導によって冷却される。電子顕微鏡鏡体７外に設けられた冷媒貯留容器１６は液体窒素貯留容器１７の内側に液体ヘリウム貯留容器１８を設け，液体窒素貯留容器に熱接触した熱シールド部材で液体ヘリウム貯留容器１８を包含して，液体窒素貯留容器１７および液体ヘリウム貯留容器１８を各々断熱支持して冷媒貯留容器１６に固定された二槽構造の冷媒貯留容器で構成する。試料１もしくは試料ホルダ２を冷却する第一冷却部材３は第二および第三の冷却部材４，５で略筒状に熱シールドされている。前記液体ヘリウム貯留容器１８に第一熱伝導部材１９の一端が結合され，この第一熱伝導部材１９が略筒状に分割され前記第一冷却部材３および第二冷却部材４に結合され冷却される。液体窒素貯留容器１７に結合された第二熱伝導部材２０は略筒状で第一熱伝導部材１９の外側に形成され第三の冷却部材５に結合され、第二冷却部材４に結合され前記第一熱伝導部材１９とともに熱シールドが行われる。このように液体窒素貯留容器１７と液体ヘリウム貯留容器１

8の二槽構造で構成することによって冷媒貯留容器16の容積を大きくすることなく液体ヘリウム貯留容器18の容積を大きくすることができる。さらに、試料1もしくは試料ホルダー2に熱伝導や輻射によって流入する外部入熱は蒸発潜熱の大きい（液体ヘリウム冷媒の約10倍）液体窒素冷媒14によって第一段階で吸収され液体窒素冷媒14の沸点より低温側は液体ヘリウム冷媒15よって吸収される。上記のことから試料1もしくは試料ホルダ2を冷却する第一冷却部材3と中間の熱シールドを行う第一冷却部材3とを液体ヘリウム貯留容器17に結合した第一熱伝導部材19の他端を略筒状に分割することで、第一冷却部材3と中間の熱シールドを行う第二冷却部材4および第一熱伝導部材19が受ける入熱を液体ヘリウム冷媒の蒸発潜熱として受け持ち第一冷却部材3と第二冷却部材4とに温度差を与えることができる。液体窒素貯留容器17および液体ヘリウム貯留容器18に結合された第一熱伝導部材19および第二熱伝導部材20の一部分を各々の冷媒容器の中に突出する事で冷媒との熱交換を容易にしている。また冷媒貯留容器16の外壁に制振材21を設け冷媒の蒸発による振動および冷媒容器外面より受ける音波等の空気振動を低減して試料への振動流入を低減する。

【0011】図3（a）は本発明の実施例で前記実施例の低温側の冷媒貯留容器に結合された熱伝導部材を二つの独立した略筒状で構成し、三層の熱伝導と熱シールドを兼ねた極低温試料冷却装置の構成図を、（b）は（a）の熱伝導部材のA－A断面図を示す。すなわち本例では、図1の実施例の液体ヘリウム貯留容器18に結合された第一熱伝導部材19を分割することなく各々独立した第一熱伝導部材22の外側に略筒状に第二熱伝導部材23を設け液体ヘリウム貯留容器18に結合する。これにより第一熱伝導部材22と第二熱伝導部材23との熱伝達系を分離することができ熱シールド特性および試料冷却到達特性を向上することができる。

【0012】図4（a）は本発明の実施例で前記実施例の低温側の冷媒貯留容器に結合された熱伝導部材を中空とし冷媒貯留容器に充填された冷媒が熱伝導部材の中空部に流入する構造の極低温試料冷却装置の構成図を、（b）は（a）の熱伝導部材のA－A断面図を示す。本例の場合は、図3の実施例の液体ヘリウム貯留容器18に結合された第一熱伝導部材22を中空とし液体ヘリウム貯留容器18に充填された液体ヘリウム冷媒15が第一熱伝導部材24の中空部に流入し液体ヘリウム冷媒15が充填されるようにした。これにより極低温試料冷却装置の大型化により冷媒貯留容器16と冷却される試料3との距離が増大し熱伝導部材（20、23、24）が長くなっても試料冷却到達特性を低下させることがない。このとき第一熱伝導部材24の中空部に僅かの傾斜をつけ冷却冷媒の気化したガスを排出し易くすることができる。

【0013】図4の実施例では第一熱伝導部材24を中空としたが同様に第二、第三の熱伝導部材20、23を中空にし熱シールド特性および試料冷却到達特性をさらに向上させることができる。

【0014】図5（a）は本発明の実施例で前記実施例の低温側の冷媒貯留容器に結合された熱伝導部材の任意の位置で熱伝導面積を可変とし、温度制御と温度補償を可能にした極低温試料冷却装置の構成図を、（b）は（a）の熱伝導部材のA－A断面図を示す。図のように、冷却された試料1もしくは試料ホルダー2の温度を高温側へ任意の温度にヒータ25等の入熱によって設定制御すると、入熱により冷却冷媒を蒸発させることとなる。冷媒の消費量を低減するため第一熱伝導部材26を分割し、分割部の熱伝導面積を可変にすることで伝熱量を制御し冷却冷媒を蒸発させることなく冷却された試料1もしくは試料ホルダー2の温度を高温側へ任意の温度に設定制御することができる。本実施例では第一熱伝導部材26の分割部にピエゾ素子27を設け、さらに、試料1もしくは試料ホルダー2の搭載された第一冷却部材3にヒータ25と温度センサー28を設け、温度制御装置29とピエゾ素子駆動装置30を閉ループで制御することで第一熱伝導部材26の分割部の熱伝導面積を可変にし、試料1もしくは試料ホルダー2の温度を任意に設定制御し温度が補償される。ピエゾ素子27によらず他の駆動手段の場合は第一熱伝導部材26に入熱がされないような駆動手段をとる必要がある。

【0015】図6（a）は本発明の実施例で前記実施例の低温側の冷媒貯留容器に結合された熱伝導部材の任意の位置で分割し、分割部熱伝導面間の熱接触時間を制御し、温度制御と温度補償を可能にした極低温試料冷却装置の構成図を、（b）は（a）の熱伝導部材のA－A断面図を示す。本図は、図5の実施例と目的を同様にした別の実施例である。冷却された試料1もしくは試料ホルダー2の温度を高温側へ任意の温度に設定制御するために、本実施例では第一熱伝導部材31の分割部にピエゾ素子32を設け、さらに試料1もしくは試料ホルダー2の搭載された第一冷却部材3にヒータ25と温度センサー28を設け、第一熱伝導部材31の分割部熱伝導面間の熱接触時間を温度制御装置29とピエゾ素子駆動装置30を閉ループで制御することで任意の温度に設定制御され温度が補償される。ピエゾ素子30によらず他の駆動手段の場合も図5の実施例と同様に、第一熱伝導部材31に入熱がされないような駆動手段をとる必要がある。

【0016】以上実施例で説明したが、実施例の略筒状に構成された熱伝導部材は製作上分割して製作し一体に結合する手段をとる場合は、熱伝導を十分考慮した手段をとる必要がある。

【0017】

【発明の効果】以上の結果、極低温試料冷却装置の冷却

到達温度を向上させるとともに、高温側への任意の温度に設定制御し温度を補償することができる。さらに、冷却冷媒の消費量、特に蒸発潜熱が小さくて高価な液体ヘリウムの消費量を削減でき、稼働コストを低減し試料冷却保持時間の向上を図ることができる。

【図面の簡単な説明】

【図１】電子顕微鏡等の従来の極低温試料冷却装置の概略構成図。

【図２】（ａ）は本発明の一実施例を示す極低温試料冷却装置の構成図、（ｂ）は（ａ）の熱伝導部材のＡ－Ａ断面図。

【図３】（ａ）は本発明の他の実施例を示す極低温試料冷却装置の構成図、（ｂ）は（ａ）の熱伝導部材のＡ－Ａ断面図。

【図４】（ａ）は本発明の他の実施例を示す極低温試料冷却装置の構成図、（ｂ）は（ａ）の熱伝導部材のＡ－Ａ断面図。

【図５】（ａ）は本発明の他の実施例を示す極低温試料冷却装置の構成図、（ｂ）は（ａ）の熱伝導部材のＡ－＊

＊Ａ断面図。

【図６】（ａ）は本発明の他の実施例を示す極低温試料冷却装置の構成図、（ｂ）は（ａ）の熱伝導部材のＡ－Ａ断面図。

【符号の説明】

１：試料、２：試料ホルダー、３：第一冷却部材、７：電子顕微鏡鏡体、８：第一液体ヘリウム貯留容器、９：第二液体ヘリウム貯留容器、１０：液体窒素貯留容器、１１：第一熱伝導部材、１２：第二熱伝導部材、１３：第二熱伝導部材、１４：液体窒素冷媒、１５：液体ヘリウム冷媒、１６：冷媒貯留容器、１７：液体窒素貯留容器、１８：液体ヘリウム貯留容器、１９：第一熱伝導部材、２０：第二熱伝導部材、２１：制振材、２２：第一熱伝導部材、２３：第二熱伝導部材、２４：第一熱伝導部材、２５：ヒータ、２６：第一熱伝導部材、２７：ピエゾ素子、２８：温度センサー、２９：温度制御装置、３０：ピエゾ素子駆動装置、３１：第一熱伝導部材、３２：ピエゾ素子。

【図１】

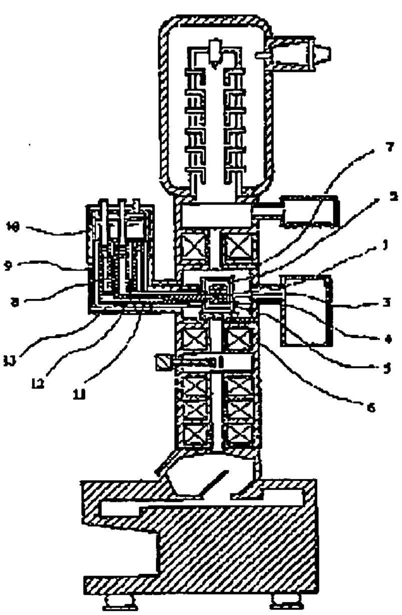


図 1

【図５】

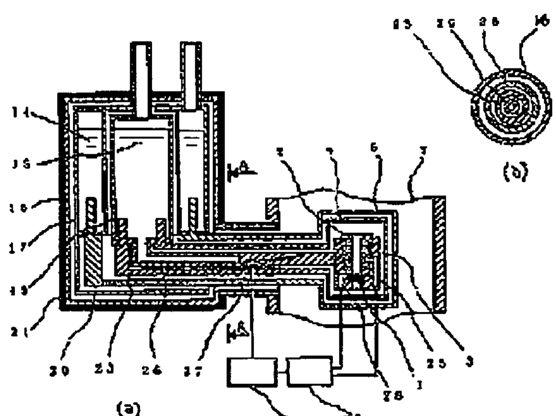


図 5

【図2】

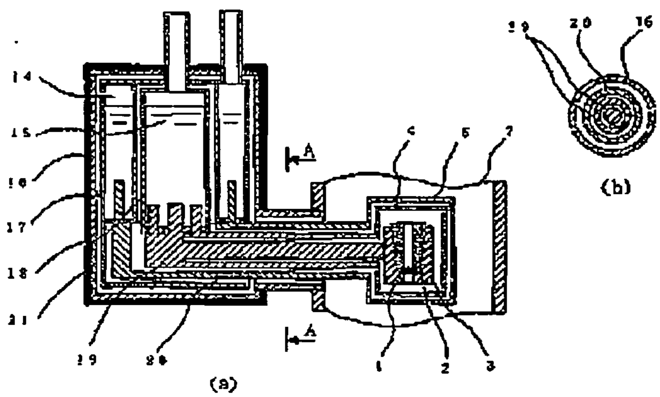

14
15
16
17
18
21
19
20
A
A
4
5
7
1
2
3
(a)
19
20
16
(b)

図 2

【図3】

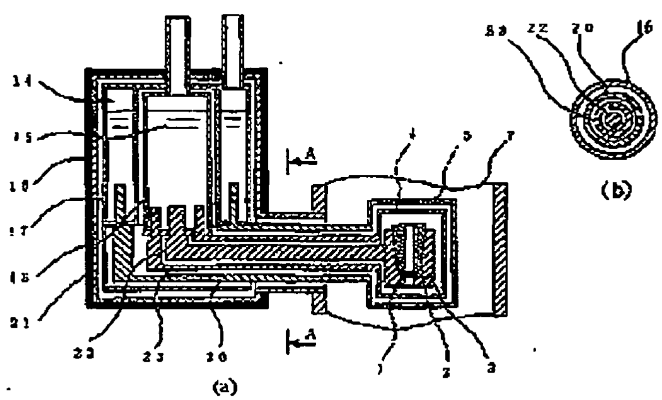

14
15
16
17
18
21
22
23
20
A
A
4
5
7
1
2
3
(a)
22
20
16
(b)

図 3

【図4】

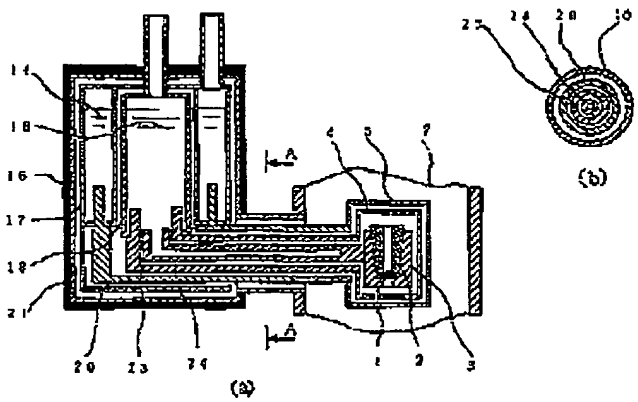


図 4

【図6】

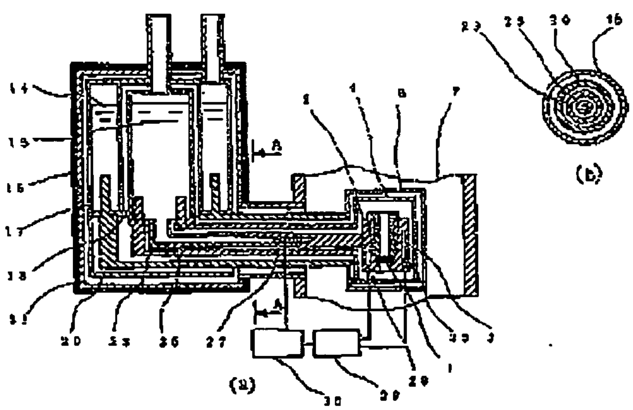


図 6

フロントページの続き

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CLAIMS

[Claim(s)]
[Claim 1] The refrigerant reservoir container for including the sample stage in which the sample or sample electrode holder installed in mirror bodies, such as an electron microscope, is carried in two or more heat-shield layers, and cooling the sample stage and each heat-shield layer, The sample cooling system characterized by considering as the heat-conduction member which the end of a heat-conduction member was combined with the refrigerant reservoir container, divided the heat-conduction section of the heat-conduction member concerned approximately cylindrical [plurality] in the sample cooling system which consists of a heat-conduction member which connects both, served as two or more heat-shield layers, and became independent respectively.
[Claim 2] association of said refrigerant reservoir container and heat-conduction member -- the sample cooling system according to claim 1 characterized by considering as the heat-conduction member which has arranged so that it may be, and the end of a heat-conduction member may be combined with a refrigerant reservoir container and the heat-conduction member concerned may be constituted from two or more approximately cylindrical heat-conduction sections, served as two or more heat-shield layers, and became independent respectively.
[Claim 3] association of said refrigerant reservoir container and heat-conduction member -- it is -- a refrigerant reservoir container -- the end of a heat-conduction member -- the inside of a refrigerant reservoir container -- a projection, a refrigerant, and a heat-conduction member -- direct contact -- the sample cooling system according to claim 1 characterized by constituting like.
[Claim 4] association of said refrigerant reservoir container and heat-conduction member -- the sample cooling system according to claim 1 characterized by constituting so that it may be, the refrigerant with which made the heat-conduction member hollow and the refrigerant reservoir container was filled up may flow into the centrum of a heat-conduction member and it may fill up with a refrigerant.
[Claim 5] The sample cooling system according to claim 1 characterized by having constituted so that the second heat-conduction member prepared in the outside of the first heat-conduction member which cools said sample or a sample electrode holder, and the first heat-conduction member approximately cylindrical might be combined with one liquid helium refrigerant reservoir container and the third heat-conduction member prepared in the outside of the second heat-conduction member approximately cylindrical might be combined with a liquid nitrogen refrigerant reservoir container, and preparing a heat-shield layer.
[Claim 6] The sample cooling system according to claim 1 or 2 characterized by controlling a heat-conduction heating value, and preparing a heat input function and a temperature detection function in a sample and a sample electrode holder or said sample, and a sample electrode-holder loading cooling member, and carrying out control compensation of the temperature of a sample or a sample electrode holder by making heat-conduction area adjustable for said heat-conduction member in the location of arbitration.
[Claim 7] The sample cooling system according to claim 6 characterized by preparing a piezo-electric element, and controlling the heat-conduction area of said heat-conduction area variant part by piezo-

electric element drive, and preparing a heat input function and a temperature detection function in a sample and a sample electrode holder or said sample, and a sample electrode-holder loading cooling member, and compensating the variant part of said heat-conduction area for the temperature of a sample or a sample electrode holder.
[Claim 8] The sample cooling system according to claim 1 or 2 characterized by dividing said heat-conduction member in the location of arbitration, and controlling the heat contact time between the division section heat-conduction sides of said heat-conduction member, and preparing a heat input function and a temperature detection function in a sample and a sample electrode holder or said sample, and a sample electrode-holder loading cooling member, and carrying out control compensation of the temperature of a sample or a sample electrode holder.
[Claim 9] The sample cooling system according to claim 7 characterized by preparing a piezo-electric element, and controlling the heat contact time between the division section heat transfer sides of said heat-conduction member by piezo-electric element drive, and preparing a heat input function and a temperature detection function in a sample and a sample electrode holder or said sample, and a sample electrode-holder loading cooling member, and compensating the division section of said heat-conduction member for the temperature of a sample electrode holder.
[Claim 10] The sample cooling system according to claim 5 prepare a liquid-nitrogen refrigerant reservoir container in the outside of said liquid-helium refrigerant reservoir container, carry out liquid-helium refrigerant reservoir container inclusion by the heat-shield member which carried out heat contact at the liquid-nitrogen refrigerant reservoir container, carry out heat insulation support immobilization at the refrigerant container which includes a liquid-helium refrigerant reservoir container and a liquid-nitrogen refrigerant reservoir container, and carry out decreasing aerial vibration, such as the acoustic wave which prepares a sound deadener in a refrigerant container periphery, and receives from the vibration and the refrigerant container external surface by evaporation of a refrigerant, as the description

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[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the very-low-temperature sample cooling system used combining the equipment which installs a sample in vacuum housings, such as an electron microscope.
[0002]
[Description of the Prior Art] As very-low-temperature sample cooling systems, such as the conventional electron microscope, the equipment in which sample cooling is possible to 5K or less very-low-temperature field is in JP,63-32847,A like a publication.
[0003] Drawing 1 shows the configuration of very-low-temperature sample cooling systems, such as the conventional electron microscope.
[0004] Install the sample stage 6 which consisted of the second [which encloses the first cooling member 3 and the first cooling member 3 which carry out heat contact with a sample 1 or the sample holder 2, and which are cooled to very low temperature approximately cylindrical, and performs a heat-shield], and third cooling members 4 and 5 in the electron microscope mirror body 7, and are mutually-independent out of the electron microscope mirror body 7. The first liquid helium reservoir container 8 which cools said first cooling member 3, the second liquid helium reservoir container 9 which cools the second [which performs a heat-shield], and third cooling members 4 and 5, and the liquid nitrogen reservoir container 10 are installed. It was combined with the first cooling section 3 and second, and third cooling member 4 and 5 by the first heat-conduction member 11 and second, and third heat-conduction member 12 and 13, respectively, and the first and second liquid helium reservoir containers 8 and 9 and the liquid nitrogen reservoir container 10 enabled cooling of a sample to very low temperature. Thus, the heating value which flows into the low-temperature section by heat conduction and thermal radiation in the conventional very-low-temperature sample cooling system The liquid nitrogen cooling member 5 cooled by about 100 K between the sample 1 and the sample electrode holder 2, and the room temperature part since it was so few that the temperature of the elevated-temperature section which contacts or faces it was low is made to intervene. 100K or more liquid nitrogen cooling members 5 and the liquid HEUMU cooling member 3 of several K have the temperature gradient which is about 100 degrees. Furthermore, in order [contact or in order to make it not face], The second liquid HEUMU cooling member 4 is made to intervene between the liquid HEUMU cooling members 3 and the liquid nitrogen cooling members 5 which cool a sample 1 and the sample electrode holder 2. It considered as the Mie heat-shield structure surrounding a sample 1 and the sample electrode holder 2, and had become the structure which cools the three above-mentioned cooling members through three heat-conduction members, respectively from three refrigerant reservoir containers. However, by this method, when a liquid nitrogen reservoir container sets change of the heating value which flows into the low-temperature section by heat conduction and thermal radiation by one tub and a liquid helium reservoir container becoming required two tubs, and sample temperature as arbitration according to the heat input of a heater etc., the latent heat of vaporization of liquid helium is as small as 1/10 compared with liquid nitrogen, and the consumption of liquid helium is greatly

influenced by the slight heat flow rate close. From this, by 2 tub methods, the conventional liquid helium reservoir container will change with experiment conditions of sample cooling of the liquid helium consumption of each reservoir container, and will be determined by the liquid helium of the side by which the time amount (sample cooling experiment) which can be sample cooled is consumed early of the liquid helium of two tubs. This cannot perform effective use of expensive liquid helium, but will consume liquid helium in large quantities, and lacks also in economical efficiency. Moreover, a reservoir container ON **** activity becomes again about liquid helium, and workability is also missing. In order that the manufacturing cost of a reservoir container might also become three reservoir containers and a refrigerant container might naturally enlarge it, there were troubles, like for a reason, a manufacture price goes up. Furthermore, aerial vibration, such as an acoustic wave received from the vibration and the refrigerant container external surface by evaporation of a refrigerant, is transmitted to a sample or a sample holder through a heat-conduction member, and the engine performance of an electron microscope etc. was reduced.

[0005]

[Problem(s) to be Solved by the Invention] In the very-low-temperature sample cooling system of the above-mentioned conventional technique, when cooling attainment temperature is determined by the cooling property of a refrigerant and a very-low-temperature sample cooling system, and it carries out setting control to an elevated-temperature side at the temperature of arbitration, and a heater etc. performs a heat input to a sample, a sample electrode holder, or the heat-conduction member to load, evaporation of a refrigerant will be brought forward and the sample cooling holding time's being shortened inevitably or an expensive refrigerant will be consumed in large quantities. For this reason, it is necessary to reduce consumption of a refrigerant and to aim at improvement in economical efficiency. Moreover, the liquid helium which the consumption of the liquid helium of two reservoir containers changed with 1:10 and small slight heat flow rate close compared with liquid nitrogen, and the sample cooling holding time was restricted by consumption of one liquid helium of the reservoir container whose number is two, and filled up two reservoir containers with the latent heat of vaporization of liquid helium is not used effectively.

[0006] Furthermore, a liquid nitrogen reservoir container needs to shorten the working hours with which one tub and a liquid helium reservoir container fill up a refrigerant in order to take great time amount to fill up a reservoir container with a refrigerant, since it has refrigerant container composition of two tubs.

[0007]

[Means for Solving the Problem] This invention makes the refrigerant reservoir container of a very-low-temperature sample cooling system 2 tub structures of a liquid-nitrogen reservoir container and the liquid-helium reservoir container in which it was prepared by that inside, prepares the heat-conduction member combined by the liquid-helium reservoir container inside the heat-conduction member combined with the liquid-nitrogen reservoir container, makes the other end of the heat-conduction section combined with this liquid-helium reservoir container the two heat-conduction sections which divided or became independent to two, and makes the heat-conduction section 3 layer structures. Furthermore, adjustable or a heat-conduction member is divided for the heat-conduction area of the heat-conduction member which cools a sample or a sample holder in respect of heat conduction, the consumption of liquid helium is restricted by controlling the heat contact time between division section heat-conduction sides, a heat input function and a temperature detection function are prepared in a sample and a sample electrode holder or said sample, and a sample electrode-holder loading cooling member, and carrying out control compensation of the temperature of a sample or a sample electrode holder is realized.

[0008] The inclusion configuration of the two heat-conduction members cooled by liquid helium inside the heat-conduction member cooled by liquid nitrogen is carried out at 3 layer structures, and a heat-shield layer is prepared and it carries out endoergic [of the heat which flows into an interlayer] by liquid helium. Furthermore, a heater and a thermo sensor are installed in the cooling member which cools a sample or a sample holder, adjustable or a heat-conduction member is divided for the heat-conduction area of a heat-conduction member in respect of heat conduction, setting control is carried out

to an elevated-temperature side at the temperature of arbitration by controlling the heat contact time between division section heat-conduction sides, and temperature is compensated. This reduces the consumption of liquid helium.

[0009]

[Embodiment of the Invention] Hereafter, one example of this invention is explained using drawing.

[0010] Projection association of the heat-conduction member is carried out in the example of this invention at the refrigerant reservoir container of 2 tub structures, and, as for (b), drawing 2 (a) shows [the other end of the heat-conduction member combined with the refrigerant reservoir container by the side of low temperature] the A-A sectional view of the heat-conduction member of (a) for two block diagrams of the very-low-temperature sample cooling system which served both as division, heat conduction of three layers, and a heat-shield approximately cylindrical. A sample 1 or the sample holder 2 carries out heat contact with this cooling system, uses liquid nitrogen 14 and liquid helium 15 for very low temperature, and is cooled by heat conduction. The refrigerant reservoir container 16 prepared out of the electron microscope mirror body 7 forms the liquid helium reservoir container 18 inside the liquid nitrogen reservoir container 17, includes the liquid helium reservoir container 18 in a liquid nitrogen reservoir container by the heat-shield member which carried out heat contact, and it constitutes with the refrigerant reservoir container of 2 tub structures which carried out heat insulation support of the liquid nitrogen reservoir container 17 and the liquid helium reservoir container 18 respectively, and were fixed to the refrigerant reservoir container 16. The heat-shield of the first cooling member 3 which cools a sample 1 or the sample holder 2 is carried out approximately cylindrical by the second and third cooling members 4 and 5. The end of the first heat-conduction member 19 is combined with said liquid helium reservoir container 18, it is divided approximately cylindrical, and said first cooling member 3 **** is combined with the second cooling member 4, and this first heat-conduction member 19 is cooled. The second heat-conduction member 20 combined with the liquid nitrogen reservoir container 17 is approximately cylindrical, and it is formed in the outside of the first heat-conduction member 19, is combined with the third cooling member 5, and is combined with the second cooling member 4, and a heat-shield is performed with said first heat-conduction member 19. Thus, the volume of the liquid helium reservoir container 18 can be enlarged, without enlarging the volume of the refrigerant reservoir container 16 by constituting from 2 tub structures of the liquid nitrogen reservoir container 17 and the liquid helium reservoir container 18. furthermore, the external heat input which flows into a sample 1 or the sample electrode holder 2 by heat conduction or radiation is absorbed on a first stage story with the large (about 10 times of a liquid helium refrigerant) liquid nitrogen refrigerant 14 of the latent heat of vaporization -- having -- the boiling point of the liquid nitrogen refrigerant 14 -- a low temperature side -- the liquid helium refrigerant 15 -- therefore, it is absorbed. By dividing approximately cylindrical, the other end of the first heat-conduction member 19 which combined with the liquid helium reservoir container 17 the first cooling member 3 which cools a sample 1 or the sample holder 2, and the first cooling member 3 which performs a middle heat-shield from the above-mentioned thing The heat input which the first cooling member 3, the second cooling member 4 which performs a middle heat-shield, and the first heat-conduction member 19 receive can be taken charge of as the latent heat of vaporization of a liquid helium refrigerant, and a temperature gradient can be given to the first cooling member 3 and the second cooling member 4. Heat exchange with a refrigerant is made easy by projecting in each refrigerant container in a part of first heat-conduction member 19 combined with the liquid nitrogen reservoir container 17 and the liquid helium reservoir container 18, and second heat-conduction member 20. Moreover, aerial vibration, such as an acoustic wave which forms a sound deadener 21 in the outer wall of the refrigerant reservoir container 16, and is received from the vibration and the refrigerant container external surface by evaporation of a refrigerant, is reduced, and an oscillating inflow in a sample is reduced.

[0011] the heat-conduction member by which drawing 3 (a) was combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention -- two -- it became independent -- it is approximately cylindrical, and constitutes and (b) shows [heat conduction of three layers, and a heat-shield] the A-A sectional view of the heat-conduction member of (a) for the

block diagram of a **** very-low-temperature sample cooling system. That is, in this example, without dividing the first heat-conduction member 19 combined with the liquid helium reservoir container 18 of the example of drawing 1 , the second heat-conduction member 23 is formed in the outside of the first heat-conduction member 22 which became independent respectively approximately cylindrical, and it combines with the liquid helium reservoir container 18. The heat transfer system of the first heat-conduction member 22 and the second heat-conduction member 23 can be separated by this, and a heat-shield property and a sample cooling attainment property can be improved.

[0012] (b) shows the A-A sectional view of the heat-conduction member of (a) for the block diagram of the very-low-temperature sample cooling system of the structure where the refrigerant with which drawing 4 (a) made hollow the heat-conduction member combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention, and the refrigerant reservoir container was filled up flows into the centrum of a heat-conduction member. In this example, the liquid helium refrigerant 15 with which made hollow the first heat-conduction member 22 combined with the liquid helium reservoir container 18 of the example of drawing 3 , and the liquid helium reservoir container 18 was filled up flows into the centrum of the first heat-conduction member 24, and it filled up with the liquid helium refrigerant 15. Even if the distance of the refrigerant reservoir container 16 and the sample 3 cooled increases and a heat-conduction member (20, 23, 24) becomes long by enlargement of a very-low-temperature sample cooling system by this, a sample cooling attainment property is not reduced. The gas which attached the slight inclination to the centrum of the first heat-conduction member 24 at this time, and the cooling refrigerant evaporated can be made easy to discharge.

[0013] In the example of drawing 4 , although the first heat-conduction member 24 was made hollow, the second and third heat-conduction member 20 and 23 can be similarly made hollow, and a heat-shield property and a sample cooling attainment property can be raised further.

[0014] (b) shows the A-A sectional view of the heat-conduction member of (a) for the block diagram of the very-low-temperature sample cooling system which drawing 5 (a) made heat-conduction area adjustable in the location of the arbitration of the heat-conduction member combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention, and made temperature control and temperature compensation possible. As shown in drawing, when setting control of the temperature of the cooled sample 1 or the sample electrode holder 2 is carried out according to the heat input of heater 25 grade to an elevated-temperature side at the temperature of arbitration, a cooling refrigerant is made to evaporate according to a heat input. Setting control of the temperature of the sample 1 cooled without dividing the first heat-conduction member 26, controlling the amount of heat transfer by making heat-conduction area of the division section adjustable, and evaporating a cooling refrigerant in order to reduce the consumption of a refrigerant, or the sample electrode holder 2 can be carried out to an elevated-temperature side at the temperature of arbitration. A piezo-electric element 27 is formed in the division section of the first heat-conduction member 26, a heater 25 and a thermo sensor 28 are further formed in the first cooling member 3 in which the sample 1 or the sample electrode holder 2 was carried, heat-conduction area of the division section of the first heat-conduction member 26 is made adjustable by controlling a temperature controller 29 and the piezo-electric element driving gear 30 by the closed loop, setting control of the temperature of a sample 1 or the sample electrode holder 2 is carried out at arbitration, and temperature is compensated with this example. It does not need to be based on a piezo-electric element 27, but, in the case of other driving means, it is necessary to take a driving means by which a heat input is not made the first heat-conduction member 26.

[0015] Drawing 6 (a) is divided in the location of the arbitration of the heat-conduction member combined with the refrigerant reservoir container by the side of the low temperature of said example in the example of this invention, the heat contact time between division section heat-conduction sides is controlled, and (b) shows the A-A sectional view of the heat-conduction member of (a) for the block diagram of the very-low-temperature sample cooling system which made temperature control and temperature compensation possible. These Figs. are the example of drawing 5 , and another example

which made the purpose the same. In order to carry out setting control of the temperature of the cooled sample 1 or the sample electrode holder 2 to an elevated-temperature side at the temperature of arbitration In this example, a piezo-electric element 32 is formed in the division section of the first heat-conduction member 31. A heater 25 and a thermo sensor 28 are formed in the first cooling member 3 in which the sample 1 or the sample electrode holder 2 was furthermore carried. Setting control of the heat contact time between the division section heat-conduction sides of the first heat-conduction member 31 is carried out at the temperature of arbitration by controlling a temperature controller 29 and the piezo-electric element driving gear 30 by the closed loop, and temperature is compensated. It is necessary to take a driving means which is not depended on a piezo-electric element 30 and by which a heat input is not made the first heat-conduction member 31 like [in other driving means] the example of drawing 5 .
[0016] Although the example explained above, the heat-conduction member constituted approximately cylindrical [an example] needs to take the means which took heat conduction into consideration enough, when taking means to divide on manufacture, to manufacture and to combine with one.
[0017]
[Effect of the Invention] While raising the cooling attainment temperature of a very-low-temperature sample cooling system the above result, setting control can be carried out and the temperature of the arbitration by the side of an elevated temperature can be compensated for temperature. Furthermore, the consumption of a cooling refrigerant, especially the consumption of liquid helium small the latent heat of vaporization and expensive can be reduced, operation cost can be reduced, and improvement in the sample cooling operating time can be aimed at.

[Translation done.]

TECHNICAL FIELD

[Field of the Invention] This invention relates to the very-low-temperature sample cooling system used combining the equipment which installs a sample in vacuum housings, such as an electron microscope.

[Translation done.]

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PRIOR ART

[Description of the Prior Art] As very-low-temperature sample cooling systems, such as the conventional electron microscope, the equipment in which sample cooling is possible to 5K or less very-low-temperature field is in JP,63-32847,A like a publication.
[0003] Drawing 1 shows the configuration of very-low-temperature sample cooling systems, such as the conventional electron microscope.
[0004] Install the sample stage 6 which consisted of the second [which encloses the first cooling member 3 and the first cooling member 3 which carry out heat contact with a sample 1 or the sample holder 2, and which are cooled to very low temperature approximately cylindrical, and performs a heat-shield], and third cooling members 4 and 5 in the electron microscope mirror body 7, and are mutually-independent out of the electron microscope mirror body 7. The first liquid helium reservoir container 8 which cools said first cooling member 3, the second liquid helium reservoir container 9 which cools the second [which performs a heat-shield], and third cooling members 4 and 5, and the liquid nitrogen reservoir container 10 are installed. It was combined with the first cooling section 3 and second, and third cooling member 4 and 5 by the first heat-conduction member 11 and second, and third heat-conduction member 12 and 13, respectively, and the first and second liquid helium reservoir containers 8 and 9 and the liquid nitrogen reservoir container 10 enabled cooling of a sample to very low temperature. Thus, the heating value which flows into the low-temperature section by heat conduction and thermal radiation in the conventional very-low-temperature sample cooling system The liquid nitrogen cooling member 5 cooled by about 100 K between the sample 1 and the sample electrode holder 2, and the room temperature part since it was so few that the temperature of the elevated-temperature section which contacts or faces it was low is made to intervene.

EFFECT OF THE INVENTION

[Effect of the Invention] While raising the cooling attainment temperature of a very-low-temperature sample cooling system the above result, setting control can be carried out and the temperature of the arbitration by the side of an elevated temperature can be compensated for temperature. Furthermore, the consumption of a cooling refrigerant, especially the consumption of liquid helium small the latent heat of vaporization and expensive can be reduced, operation cost can be reduced, and improvement in the sample cooling operating time can be aimed at.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] In the very-low-temperature sample cooling system of the above-mentioned conventional technique, when cooling attainment temperature is determined by the cooling property of a refrigerant and a very-low-temperature sample cooling system, and it carries out setting control to an elevated-temperature side at the temperature of arbitration, and a heater etc. performs a heat input to a sample, a sample electrode holder, or the heat-conduction member to load, evaporation of a refrigerant will be brought forward and the sample cooling holding time's being shortened inevitably or an expensive refrigerant will be consumed in large quantities. For this reason, it is necessary to reduce consumption of a refrigerant and to aim at improvement in economical efficiency. Moreover, the liquid helium which the consumption of the liquid helium of two reservoir containers changed with 1:10 and small slight heat flow rate close compared with liquid nitrogen, and the sample cooling holding time was restricted by consumption of one liquid helium of the reservoir container whose number is two, and filled up two reservoir containers with the latent heat of vaporization of liquid helium is not used effectively.
[0006] Furthermore, a liquid nitrogen reservoir container needs to shorten the working hours with which one tub and a liquid helium reservoir container fill up a refrigerant in order to take great time amount to fill up a reservoir container with a refrigerant, since it has refrigerant container composition of two tubs.

[Translation done.]

MEANS

[Means for Solving the Problem] This invention makes the refrigerant reservoir container of a very-low-temperature sample cooling system 2 tub structures of a liquid-nitrogen reservoir container and the liquid-helium reservoir container in which it was prepared by that inside, prepares the heat-conduction member combined by the liquid-helium reservoir container inside the heat-conduction member combined with the liquid-nitrogen reservoir container, makes the other end of the heat-conduction section combined with this liquid-helium reservoir container the two heat-conduction sections which divided or became independent to two, and makes the heat-conduction section 3 layer structures. Furthermore, adjustable or a heat-conduction member is divided for the heat-conduction area of the heat-conduction member which cools a sample or a sample holder in respect of heat conduction, the consumption of liquid helium is restricted by controlling the heat contact time between division section heat-conduction sides, a heat input function and a temperature detection function are prepared in a sample and a sample electrode holder or said sample, and a sample electrode-holder loading cooling member, and carrying out control compensation of the temperature of a sample or a sample electrode holder is realized.
[0008] The inclusion configuration of the two heat-conduction members cooled by liquid helium inside the heat-conduction member cooled by liquid nitrogen is carried out at 3 layer structures, and a heat-shield layer is prepared and it carries out endoergic [of the heat which flows into an interlayer] by liquid helium. Furthermore, a heater and a thermo sensor are installed in the cooling member which cools a sample or a sample holder, adjustable or a heat-conduction member is divided for the heat-conduction area of a heat-conduction member in respect of heat conduction, setting control is carried out to an elevated-temperature side at the temperature of arbitration by controlling the heat contact time between division section heat-conduction sides, and temperature is compensated. This reduces the consumption of liquid helium.
[0009]
[Embodiment of the Invention] Hereafter, one example of this invention is explained using drawing.
[0010]

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] The outline block diagram of the conventional very-low-temperature sample cooling systems, such as an electron microscope.
[Drawing 2] For (a), the block diagram of the very-low-temperature sample cooling system in which one example of this invention is shown, and (b) are the A-A sectional view of the heat-conduction member of (a).
[Drawing 3] For (a), the block diagram of the very-low-temperature sample cooling system in which other examples of this invention are shown, and (b) are the A-A sectional view of the heat-conduction member of (a).
[Drawing 4] For (a), the block diagram of the very-low-temperature sample cooling system in which other examples of this invention are shown, and (b) are the A-A sectional view of the heat-conduction member of (a).
[Drawing 5] For (a), the block diagram of the very-low-temperature sample cooling system in which other examples of this invention are shown, and (b) are the A-A sectional view of the heat-conduction member of (a).
[Drawing 6] For (a), the block diagram of the very-low-temperature sample cooling system in which other examples of this invention are shown, and (b) are the A-A sectional view of the heat-conduction member of (a).
[Description of Notations]
A sample, 2:sample electrode holder, the 3:first cooling member, 7 : 1: An electron microscope mirror body, 8: The first liquid helium reservoir container, the 9:second liquid helium reservoir container, 10 : A liquid nitrogen reservoir container,

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DRAWINGS

[Drawing 1]

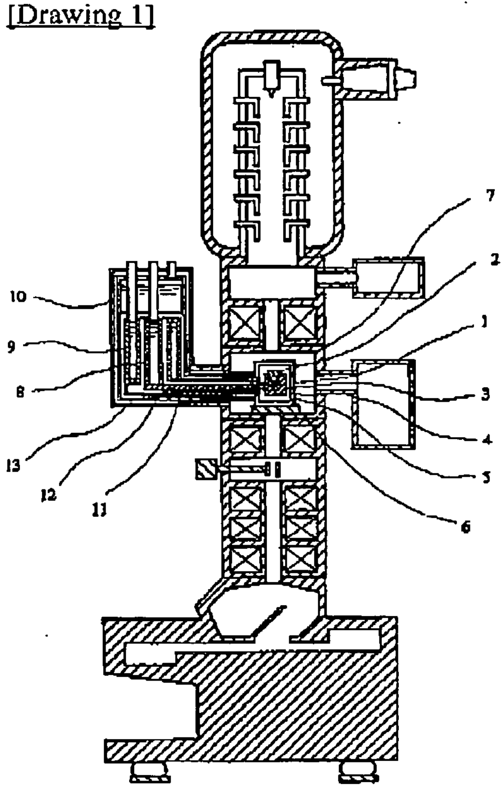

図 1

[Drawing 5]

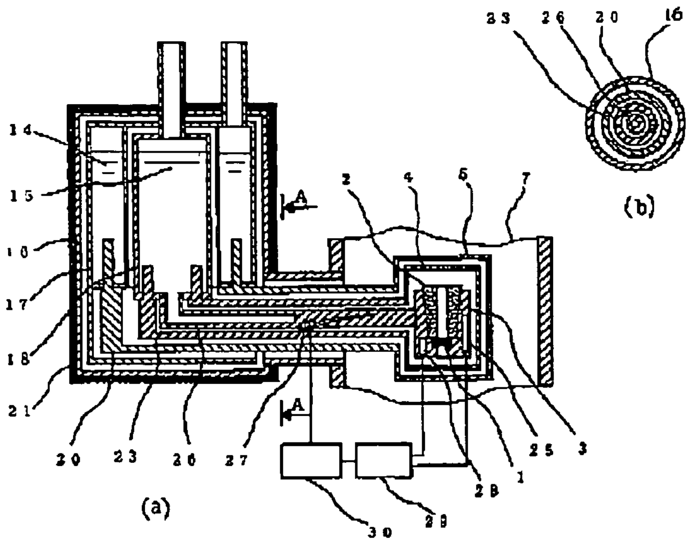

図 5

[Drawing 2]

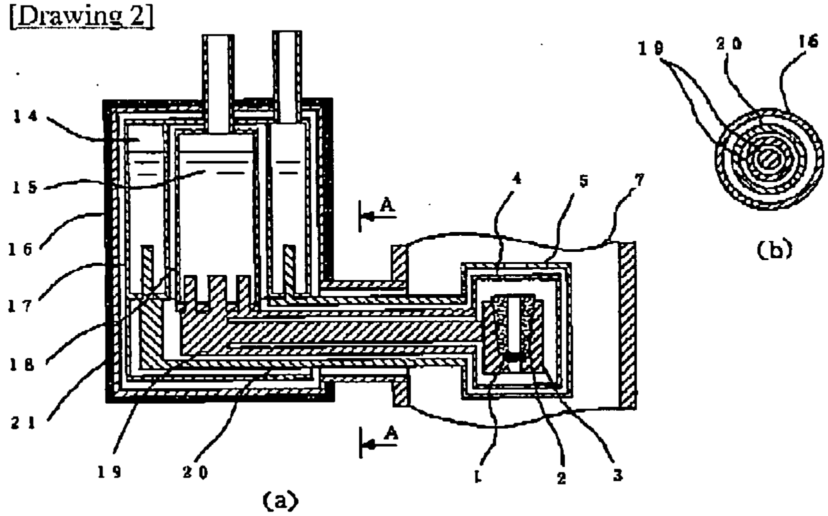

図 2

[Drawing 3]

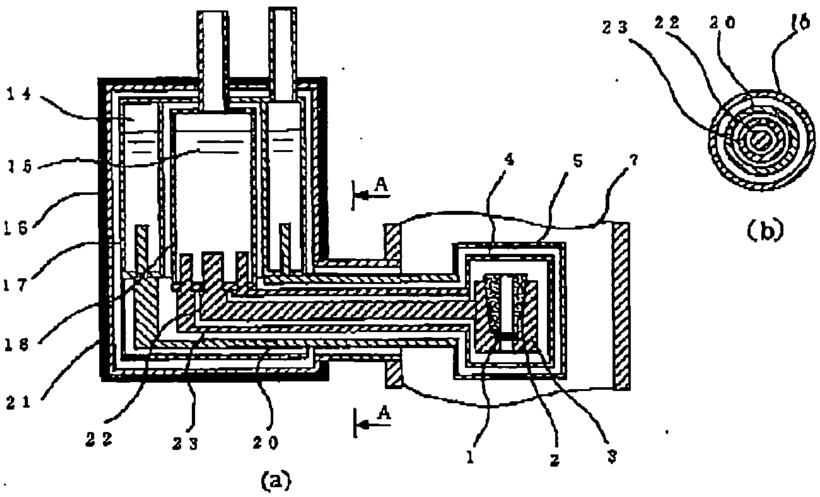

図 3

[Drawing 4]

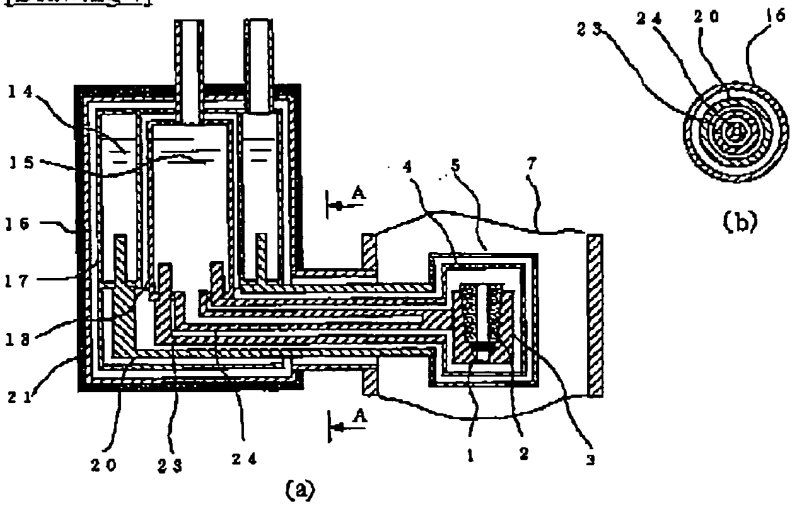

図 4

[Drawing 6]

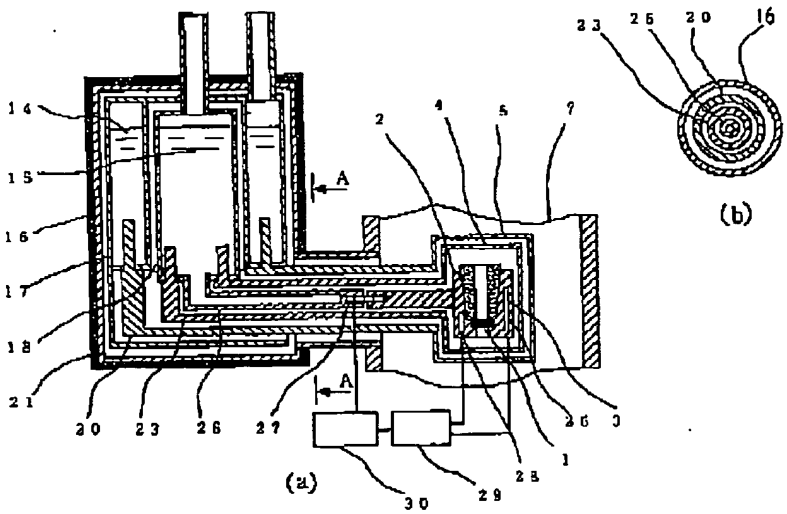

図 6

[Translation done.]